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# NASA TECHNICAL MEMORANDUM

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CHEMICAL VAPOR DEPOSITION FOR AUTOMATIC  
PROCESSING OF INTEGRATED CIRCUITS

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# CHEMICAL VAPOR DEPOSITION FOR AUTOMATIC PROCESSING OF INTEGRATED CIRCUITS

## PART I: CHEMICAL VAPOR DEPOSITION

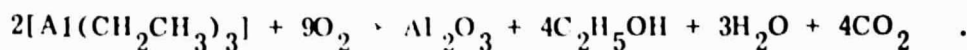
### A. INTRODUCTION

Passivation methods for hermetic sealing of semiconductor devices are generally divided into vapor plating, vacuum, and organic techniques. Vapor plating techniques for depositing various types of glass films for semiconductor passivation applications are the most successful passivation methods related to integrated circuits. Therefore, most of the data presented herein concerns vapor plating-techniques. Passivation using electron beam deposited quartz is briefly discussed.

### B. VAPOR PLATING TECHNIQUES

Vapor plating is a process in which a gas compound or gas compound mixtures react at a surface to deposit a low vapor pressure film. Thermal decomposition of single compounds in the vapor state is more commonly referred to as pyrolysis. The basic criteria for vapor plating to occur are (1) the starting reactants must be in the vapor state, (2) the primary reaction must take place at a designated surface, (3) the product of the reaction must be stable at the reaction temperature, and (4) any byproducts of the reaction should possess a high vapor pressure to prevent contamination of the low vapor pressure deposit.

Vapor plating as used for the passivation of semiconductor devices is illustrated by the mixing of an aluminum alkyl with  $O_2$  or an oxygen-containing compound to form an amorphous film of  $Al_2O_3$  at a surface held at an elevated temperature. The primary reactions that participate in this vapor-plating process are:



From these basic reactions, numerous vapor-plated compounds have been developed and used for the passivation and product improvement of semiconductor devices.

A composition glass is formed by using aluminum alkyl in conjunction with tetraethyl orthosilicate (as a silica source) and/or tri-isopropyl orthosilicate (as a silica source) and/or tri-isopropyl borate (as a boron oxide source). The combination of these chemicals has produced the most successful low temperature oxide in use to date. This compound designated as  $B_2O_3 \cdot Al_2O_3 \cdot SiO_2$  and called boroaluminosilicate combines many highly desirable dielectric properties into a single glass film which could only be formed from the vapor state. The deposition temperature of 300° to 400°C allows high compatibility with almost all current semiconductor technology.

Vapor-plating techniques fall into two broad categories which are based on the source used for silicon.

1. Ethyl Silicate Systems. Ethyl silicate systems use tetraethyl orthosilicate (ethyl silicate) as the silica source. These systems are limited in their applications by the high substrate temperatures required and by slow deposition rates.

In the early process, research and development of vapor plated dielectric films was carried out with various one-component systems; from these one-component glasses, several binary and ternary glasses evolved. The various glasses which have been used for passivating agencies are:

$SiO_2$	Silica
$Al_2O_3$	Alumina
$Al_2O_3 \cdot SiO$	Aluminosilicate
$B_2O_3 \cdot Al_2O_3 \cdot SiO_2$	Boroaluminosilicate
$B_2O_3 \cdot SiO_2$	Borosilicate

A single deposition system is capable of producing any one of these glass films. The major vapor-deposited glass film now used is boroaluminosilicate. This film has been evaluated under numerous types of environmental and long-term reliability tests with different processing variables. It was widely used during its development in all phases of semiconductor technology.

2. Silane Systems. Another vapor-deposited glass film is based on the use of  $SiH_4$  as the source for silicon. The more chemically reactive  $SiH_4$  produces hard and adherent films of high purity  $SiO_2$  at very moderate temperatures. The apparatus for deposition of  $SiO_2$  films from  $SiH_4$  consists of a reaction chamber in which the reactive  $SiH_4$  is synthesized in a controlled manner, a plating head in which the vaporized silicon compound is mixed with oxidizing gases, and a substrate heater which supports the substrate below the plating head and maintains the substrate

at a desired deposition temperature. The major advantage of the  $\text{SiH}_4$  system depositions is the ability to reach deposition rates in the range of 2000 and 5000 Å per minute, with the possibility of lower substrate temperatures.

### C. BOROSILICATE SILICA GLASSING

In the manufacture of semiconductor transistors and integrated circuits, it is necessary to provide protection against contaminants that can degrade the electrical characteristics of the devices. Moisture is considered the main agent which must be kept away from the surface. For silicon devices, it is common practice to provide a passivating coating of  $\text{SiO}_2$ , usually by thermally oxidizing the surface of the silicon. Thermally grown  $\text{SiO}_2$  imparts to the device surface a remarkable degree of passivation but it is not always adequate to preserve initial device characteristics for an indefinite time and under a variety of ambients. Its molecular network structure is relatively open and not impermeable to contaminants. Thick layers of  $\text{SiO}_2$  lack mechanical strength because of the thermal expansion mismatch with the substrate. In addition, the layer must be open to permit contact to be made to the device. These open areas are particularly susceptible to contamination from the ambient. Accordingly, oxide-protected devices are sealed in metal cans or in ceramic flat packs. Sometimes, at some risk, they may be embedded in polymeric plastic materials. Both of these encapsulation techniques have disadvantages. Metal can structures and ceramic packages are expensive and occupy such large volumes that much of the potential advantage of small size provided by integrated circuit technology is not realized. Plastic encapsulants, under certain situations, are not impervious to contaminants. Moreover, these materials often act as, or contain, contaminants themselves.

Silicate glasses are recognized as a solution to most of these encapsulation problems. Various methods of applying glass coatings to the surface of semiconductor devices have been proposed but have met with only limited success. For example, fusion techniques have been developed in which the glass is applied to the device surface as a frit that is heated to a temperature above its softening point. The temperatures required for applying the coatings by these methods are often so high that they are restricted to special high temperature metallization systems or to glassing prior to metallization which must then be of limited thickness so that patterns can be etched in these with sufficient precision. Special low temperature glasses have been derived for glassing conventionally metallized silicon devices, but they are more or less moisture sensitive. Other known techniques for forming glass layers include reactive sputtering and RF sputtering, but these generally must be restricted to simple glass compositions and the deposition rates are low. Furthermore, annealing after deposition is generally required if dense, impermeable films are to be obtained.

Recent efforts in industry have been concentrated on developing a glassing technology free from these various drawbacks. The objective was to devise a batch process, based on chemical vapor deposition at low temperatures, for hermetically glassing semiconductor devices in wafer form. This type of glassing can be considered a key to a simple and much less expensive package because the glass layer serves as the hermetic seal and the costly sealed metal cans and ceramic flat packs are no longer required.

The individual metallized chips can be attached to an inexpensive header in conventional fashion, or they can be mounted face down either on a suitable fanout pattern or directly on a printed circuit board. The completed transistor or integrated circuit package might, for instance, be coated with a plastic polymer to afford mechanical protection to the package shield it from light, and to act as a first barrier against gross contamination.

Inexpensive plastic and ceramic substrate holders with metallization fanouts have been developed on which the glassed chips can be mounted by solder reflow or ultrasonic bonding techniques. Thus, all contacts on a chip can be made simultaneously in one operation, in contrast to the individual wire bonding of each contact now conventionally used.

In addition to the primary purpose of chemically vapor-deposited glass films (i.e., to provide an impermeable insulating and passivating coating), they are useful as an air isolation layer in preparing crossover patterns. As circuit design becomes more sophisticated, these and other applications of vapor glassing will become essential, especially for computer arrays of higher device speeds in high density with improved reliability and low costs.

#### D. GLASS DEPOSITION

Glass deposition from the vapor phase by chemical reaction has the following distinct advantages over the previously described methods:

- 1) A relatively low substrate temperature is required for film deposition. The glass compositions are synthesized well below their softening temperature. This low-temperature feature is important because changes in diffusion profiles are prevented or minimized, deleterious effects such as oxidation of the metal leads are avoided, and impurities on the surface are prevented from diffusing into the interior of the device.

- 2) A wide choice of compositions of high purity can be prepared by controlled variation of the vapor composition. The compositions can be tailor-made to provide desirable properties, such as a matching thermal expansion coefficient. Glass compositions of high purity can readily be made because the reagents used are of high purity, and contamination during deposition and subsequent handling can be made negligible.

3) Integral films with gradually or abruptly changing compositions can be prepared in one deposition operation simply by changing the reactant vapors introduced into the reaction chamber.

4) The film thickness can be readily controlled. For example, on silicon substrates, glass thicknesses ranging from 500 to 250,000 Å have been reproducibly deposited.

5) The film thickness is highly uniform over the exposed surface of a device, regardless of its shape. Furthermore, film deposits are virtually free of pinholes and cracks. The films can also be deposited in selected areas of a wafer through appropriate masks.

6) The processes for depositing the films are economical and practical because fairly high film growth rates can be achieved. The deposition equipment is relatively simple and can be scaled up for large scale batch production.

7) The dielectric and chemical properties of the glass films and their hermeticity can be additionally improved by a brief heat treatment at a temperature above the deposition temperature. The delineation of patterns by photomasking and etching methods may be carried out on undensified layers which exhibit greater solubility than densified films.

8) The adherence of vapor-deposited glass films to metallic surfaces, such as tungsten metallization on devices, is much better than for glass films deposited by fusion techniques. The quality of the glass-to-metal seal should, therefore, be superior.

The principal disadvantage of the chemical vapor glassing process is the care required to control the proper reagent flow rates. If they vary, improper composition of the film results. A second disadvantage is that the reactants are toxic and flammable.

Two main types of chemical vapor glassing have been developed and are distinguished as follows:

1) High temperature (700° to 800°C) reactions with organo-metallic reagents employing an open furnace.

2) Low temperature reactions (275° to 475°C) with inorganic reagents in a rotary (hot plate) reactor.

The high temperature processes are based on organo-silicon compounds, such as tetraethoxy silane, as sources for the silicon component in the glasses, whereas the low temperature processes use  $\text{SiH}_4$  to provide the silicon.

The  $\text{SiH}_4$ -based, low temperature systems have considerable advantages over the older type of systems, and have, therefore, been developed to a much greater degree of refinement.

The most satisfactory compositions developed for silicon device glassing are:

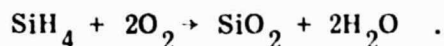
1) The borosilicate compositions synthesized from  $\text{SiH}_4$ , diborane ( $\text{B}_2\text{H}_6$ ), and  $\text{O}_2$  which are readily prepared and are satisfactory for most purposes of device glassing.

2) The aluminoborosilicates from  $\text{SiH}_4$ ,  $\text{B}_2\text{H}_6$ , trimethyl aluminum, and  $\text{O}_2$  which are more difficult to prepare but offer improved chemical stability.

3) Laminates consisting of combinations of borosilicates or aluminoborosilicates and  $\text{SiO}_2$ , with or without base layers of  $\text{Si}_3\text{N}_4$ .

Various processes of glass deposition from the vapor state are discussed. Included are  $\text{SiO}_2$ , borosilicates, aluminosilicates, phosphosilicates, and laminates.

1. Silicon Dioxide.  $\text{SiO}_2$  is the most commonly used single component glass. It can be deposited by the thermal decomposition of  $\text{SiH}_4$  with  $\text{O}_2$  at a preferred temperature in the range of  $450^\circ$  to  $475^\circ\text{C}$ . Lower temperatures (down to  $250^\circ\text{C}$ ) can be used, but deposition rate and film quality decrease. Although  $\text{SiO}_2$  gives a remarkable degree of passivation, its relatively open molecular structure is not sufficiently closed to contaminants. For this reason,  $\text{SiO}_2$  passivated devices must be sealed in either metal or ceramic packages. Thick layers have little mechanical strength due to the thermal mismatch between  $\text{SiO}_2$  and the substrate. The reaction for thermally deposited  $\text{SiO}_2$  from  $\text{SiH}_4$  and  $\text{O}_2$  is



2. Borosilicates. Borosilicates (binary component glasses) are deposited from the reaction of  $\text{SiH}_4$ ,  $\text{B}_2\text{H}_6$ , and  $\text{O}_2$  at  $450^\circ\text{C}$ . Compositions with different thermal expansion coefficients, softening points, etch rates, etc., are possible. For example, compositions obtained from 20 to 25 volumes of pure  $\text{B}_2\text{H}_6$  and 75 to 80 volumes of pure  $\text{SiH}_4$  are most suitable for silicon device passivation. Compositions obtained from 25 to 30 volumes of  $\text{B}_2\text{H}_6$  and 70 to 75 volumes of  $\text{SiH}_4$  give the best thermal expansion match for silicon. These films are similar to the commercial Pyrex type of borosilicate glasses but they contain no alkalis and are of higher purity. Films up to  $25\text{ }\mu\text{m}$  thick have been deposited without defect formation.

Deposition rates up to  $2400\text{ }\text{\AA}$  per minute have been reached, although the rate of  $5\text{ to }6\text{ }\mu\text{m}$  per hour is preferred.



3. Aluminosilicates. The aluminosilicates (ternary component glasses) are deposited from the reaction of  $\text{SiH}_4$ ,  $\text{B}_2\text{H}_6$ , trimethyl aluminum, and  $\text{O}_2$ . They have excellent chemical and physical properties. The  $\text{Al}_2\text{O}_3$  gives the glass a greater stability and inertness than the borosilicates. This glass is not as clean as the borosilicates, but etches at similar rates and has excellent dielectric and hermetic properties.

4. Phosphosilicates. Phosphosilicates [prepared from phosphine ( $\text{PH}_3$ )  $\text{SiH}_4$ , and  $\text{O}_2$ ] are hygroscopic and do not densify with heat treatment. They do have some use in laminates.

5. Laminates. The most commonly used laminate is a layer of  $\text{SiO}_2$  over a thick layer of either borosilicate, aluminoborosilicate, or phosphosilicate glass. The most useful is  $\text{SiO}_2$  over a borosilicate.

#### E. THERMAL DENSIFICATION OF GLASS FILMS

The glass films deposited at the usual low temperatures have remarkably good properties and are satisfactory for certain applications, but a distinct enhancement of the film properties can be achieved by a brief heating at an elevated temperature to form dense, stable, and more chemically resistant glasses. This effect appears to be based primarily on a change in the bonding between the silicon-oxygen linkages, resulting in a consolidation of the molecular structure. The effects of these changes can be observed in the infrared spectra, chemical stability, dielectric breakdown strength, density, and especially the dissolution rate which decreases sharply. Most glass films deposited by chemical vapor reactions and RF sputtering techniques, including the  $\text{SiO}_2$  and  $\text{Al}_2\text{O}_3$  films, have been found to undergo densification except the films deposited from mixtures of  $\text{SiH}_4$  and  $\text{PH}_3$ . The effects of densification on  $\text{SiO}_2$  films have recently been measured in some detail by several investigators.

The degree of densification of most deposited glass films is a function of temperature, time, and ambience.  $\text{O}_2$  exerts an accelerating effect. Temperatures below the softening point of the glass should be chosen to retain the integrity of the film structure. At a given temperature, densification begins rapidly and then tapers off until changes become negligibly slow.

#### F. SUBSTRATE MATERIALS FOR GLASSING

The glass synthesis processes that have been described are applicable for coating of a wide range of substrate materials. The properties of the glasses are readily adjustable by changing the vapor composition, making it possible to tailor make coatings with thermal expansion properties

that closely match those of the substrates. The thicker the coating that is required, the better the thermal expansion match must be. Films up to a few thousand angstroms thick need not match closely, especially if the expansion coefficient of the film is lower than that of a much thicker substrate, i.e., the film is under compression.  $\text{SiO}_2$  films on silicon are a good example.

Another requirement for successfully glass-coating a given substrate material is that it must be thermally and chemically stable under the glassing conditions. Aluminum reacts slightly with borosilicates and strongly with phosphosilicates during glassing reactions that employ the hydrides, which necessitates the deposition of a thin protective layer of  $\text{SiO}_2$  preliminary to the binary glass deposition to prevent the formation of difficult soluble interfaces that may interfere in metallurgical bonding.

The following materials have been successfully glassed using the standard rotary reactor glassing techniques: silicon ceramic compositions, fused quartz plates, silicate glass plates, silicon nitride, ferrites, alumina, tungsten, molybdenum, Kovar, nickel, copper, silver, chromium, steel, aluminum, platinum, and gold.



## PART II: CHEMICAL VAPOR DEPOSITION OF DIELECTRIC FILMS FOR INTEGRATED CIRCUIT

### A. INTRODUCTION

The rapid development of the semiconductor industry over the last decade has placed great demands on dielectric and metallization technology since, to a large extent, it controls the technological pace of the electronics industry. This demand has challenged the thin-film technologist to develop new and improved processes for the thin-film conductors and insulation required for semiconductor devices. The variety of materials and processes required to adequately meet the total needs of the industry has necessitated the development of several deposition technologies. Vacuum evaporation, sputtering, chemical vapor deposition, sedimentation, etc., are all in volume manufacturing use, and the technologies of each of these techniques have been significantly improved during the past 10 years.

Perhaps the most important reason for the widespread use of thin films in microelectronics is that only thin films can be processed to yield the small size, low power, and high circuit density desired. Thin films are important in other essential processes, e.g., diffusion masking. Also, thin-film deposition techniques allow convenient production of high-purity substances or materials with closely controlled composition. Finally, some vital features of present circuits can be achieved only by using thin films, such as silicon surface passivation with  $\text{SiO}_2$ . Most thin-film applications require either highly conductive or insulating materials, although resistive and semiconductive films have their uses. These films serve as the basis of passive elements, interconnect active elements, protect and insulate various portions of the circuit, connect it to external circuitry, and furnish the active medium of the semiconductor itself. Thin films are used as functional elements of the completed device and are essential in the fabrication of the device.

Contained herein are brief descriptions of film deposition methods and the characteristics of various dielectric and metal films. Among the dielectrics described are silicon nitride ( $\text{Si}_3\text{N}_4$ ); silicon deposited from dichlorosilane ( $\text{SiH}_2\text{Cl}_2$ ), aluminum oxide ( $\text{Al}_2\text{O}_3$ ) and  $\text{Si}_3\text{N}_4$  hermetic seal layers; and thermally grown  $\text{SiO}_2$ ; borosilicate, aluminosilicate, phosphosilicate glasses and laminates of these glasses by chemical vapor deposition are also included. Metallization for monolithic circuits includes descriptions of the multilayer metal process, effect of substrate temperature on film deposition and metal sputtering on MOS devices. The characteristics of thin films and beam leaded silicon on sapphire are also discussed.

Dielectric thin films are widely used in microelectronics. These films can be deposited by chemical reaction, chemical vapor deposition (CVD), vacuum evaporation, or sputtering techniques. Chemical reaction relies on a chemical reaction between the original surface and its environment. CVD uses either a chemical reaction between two or more substances or a chemical decomposition. Vacuum evaporation occurs when the chamber pressure is reduced to the point where the substance will evaporate. These vapor pressures range from 1 to  $10 \times 10^{-3}$  torr in the temperature range of 600° to 1200°C. Sputtering is a technique in which the material ejected from a bulk source by ion bombardment is used to form the film.

Beam lead chips with silicon nitride passivation offer a tremendous potential for packaging semiconductor circuits. Silicon nitride passivation makes the circuits as failure resistant as conventional circuits in hermetically sealed packages. The silane ammonia-hydrogen reaction is satisfactory for the deposition of silicon nitride. Ammonia and silane are reacted in the presence of excess hydrogen at about 900°C in an RF-heated horizontal-tube reactor.

Dichlorosilane is an ideal source material for epitaxial silicon film growth. Its low pressure gaseous nature provides convenient delivery to the epitaxial system and low reaction temperatures.

Aluminum oxide may be deposited by the plasma oxidation of aluminum, pyrolysis of aluminum isopropyl oxide, or the hydrolysis of aluminum chloride. The hydrolysis of aluminum chloride is most used for complementary MOS beam lead devices. The aluminum oxide layers produced by this method have reproducible physical and electrical properties.

CVD processes for synthesizing glass films on semiconductor wafers fall into two basic types: (1) systems based on high temperature (700° to 800°C) reactions using organo-metallic reactants and (2) systems based on low temperature (275° to 475°C) reactions of hydrides and metal alkyls with oxygen. Glass laminate structures with either graded or abrupt multiple layers can be designed to serve specific purposes in device glassing. Glasses of various properties can be obtained by varying the vapor composition. It is possible, for example, to optimize the thermal expansion of the glass to match a given substrate. Properties of CVD glass films can be substantially improved either by a brief heat treatment (at a temperature above that used in deposition) or by a prolonged heat treatment at the temperature of deposition, which can be as low as 450°C. Numerous substrate materials can be glassed. Among these materials are silicon, germanium, gallium arsenide, ferrites, ceramics, and a host of metals. The borosilicate compositions synthesized from diborane, silane, and oxygen at a temperature of 450°C in the rotary reactor apparatus are the most attractive of the binary compositions for device glassing, especially if an over-layer of silicon dioxide is present. They are readily deposited and can be synthesized to closely match the thermal expansion of silicon. Films up to 24- $\mu$ m thickness have been deposited on silicon without defect formation. Deposition rates up to 2400 Å per minute have been achieved. The

aluminoborosilicate compositions synthesized from trimethyl aluminum, diborane, silane, and oxygen in the rotary reactor have excellent chemical and physical properties. Their composition is more difficult to control than the binary borosilicates and they tend to be less clear. Excellent results have been obtained with lead borosilicate glasses synthesized from tetraethyl lead, trimethyl borate, tetraethoxy silane, and oxygen in a horizontal tube furnace reactor at 730°C. This process is particularly useful for applications where thin films of high density are required, such as in glassing devices prior to metallization.

One solution to the problems brought about by increasing die size is to use more than one layer of metallization. Two- and three-layer parts can be manufactured reliably with acceptable yields in an aluminum-based system with phosphorosilicate glass passivation. Coverage with both metal and oxide is a prime consideration. Although single-layer parts have some coverage problems, the variety of steps encountered in the multilayer circuit make the problems more acute.

Important among the factors determining metal coverage is the specific coating system with which metal depositions are made. Even with the most sophisticated designs, the use of increased wafer temperatures and deposition rates appears to be essential to good metal coverage. Step profiling can result in ideal coverage even with nonideal deposition control. Electromigration failures must be compensated for in the design if beveled metallization is employed.

Oxide coverage is important for two reasons. Edge profiles are dependent upon oxide-to-metal thickness ratios and can often be cusped causing almost impossible conditions for metal step coverage. Pinhole protection between two metal layers depends upon how the oxide covers its underlying surface.

## B. METHODS OF FILM DEPOSITION

Many methods are available for forming surface films, and most of them are not recent discoveries. For example, sputtering was observed in the mid-19th century and, vacuum evaporation was an annoyance to the early incandescent lamp manufacturers. It will be seen that, within each general type of film deposition method, there is a wide range of materials, properties, and applications.

1. Chemical Reaction. The chemical reaction between the original surface and its environment can produce a thin layer of a new substance on the surface. The basis of the present technology of silicon devices and integrated circuits in the formation of a thin film of  $\text{SiO}_2$  on silicon is given by the following reaction:

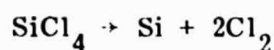
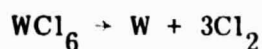
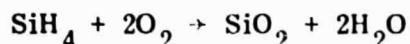


The resulting amorphous  $\text{SiO}_2$  layer serves to passivate the surface of the silicon. It also functions as a convenient diffusion mask and provides the electrical insulation between the silicon and the interconnect lines. The  $\text{SiO}_2$  layer can be formed in a number of different oxidizing gases, notably steam ( $\text{H}_2\text{O}$ ), nitrous oxide ( $\text{N}_2\text{O}$ ) and, to a lesser extent, in carbon dioxide ( $\text{CO}_2$ ). Primarily, the reaction is controlled by the rate of diffusion of oxidant across the  $\text{SiO}_2$  film; hence, the growth rate decreases with time.

No other common semiconductor forms a naturally occurring oxide film which has useful properties. To a great extent, this is why silicon is predominant in integrated circuit applications.

In thermal oxidation, the oxidant diffuses through the growing film to the oxide-silicon interface. There is another class of reactions in which the opposite reaction occurs: The silicon (or conductor) cation migrates toward and reacts with the oxidizing species at the outer surface. It is common to assist the process by making the conductor electrically positive with respect to its surroundings. This is then called anodization and is widely practiced to yield thin oxide dielectric films on metals. It can also be used to produce films on silicon.

2. Chemical Vapor Deposition. Chemical vapor deposition (CVD) employs either a chemical reaction between two or more species or a chemical decomposition to produce the desired film. The latter method is somewhat imprecisely referred to as pyrolysis. In these reactions, the surface does not play an active role in the formation of the thin film. The types of thin films that can be deposited by this method are exemplified by the following reactions:



The third chemical reaction is of particular importance. Depending on the surface conditions and crystal structure, the resulting silicon film can be single crystal or polycrystalline in nature. The former process is called epitaxy and is not usually considered a thin-film process, although most epitaxial silicon films are much less than 5  $\mu\text{m}$  thick and fall within the physical classification of thin film. If the film is polycrystalline, as it is on  $\text{SiO}_2$ , then this process is definitely a thin-film process. It is widely used to form the gate electrode in the silicon self-aligned gate FET process.

A practical CVD process requires a suitable compound to carry the desired substances to the surface, a heterogeneous gas-solid reaction whose rate is much faster than any competing process and a reactor system which assures temperature uniformity, gas composition homogeneity, and a reasonable batch size. Temperature control is important since the reaction is usually thermally activated. However, the substrate temperatures needed for film deposition may be lowered appreciably by supplying RF or microwave energy to create a plasma or glow discharge. Films of  $\text{SiO}_2$  and  $\text{Si}_3\text{N}_4$  are often deposited in this way, but reproducible film properties are sometimes difficult to achieve.

The deposition of  $\text{SiO}_2$  is one of the most widely used CVD processes. In addition to pure  $\text{SiO}_2$ , processes have been developed to incorporate stabilizing substances into the basic  $\text{SiO}_2$  matrix; glassy materials containing phosphorus, aluminum, boron, etc., have been deposited as thin films. In addition to polycrystalline silicon, a number of thin metal films can be formed by CVD. Both molybdenum and tungsten can be deposited by decomposing their halides or carbonyl compounds.

# PART III. THE DEVELOPMENT OF AN AUTOMATIC SYSTEM FOR CHEMICAL VAPOR DEPOSITION (CVD) FOR INTEGRATED CIRCUITS

## A. INTRODUCTION

A complete CVD system has been developed in accordance with the following requirements: using 3 in. silicon wafers, 3 in. sapphire substrates or 3 in. spinel substrates. Figure 1 presents a typical continuous CVD system block diagram. All process parameters are controlled by a programmer that can be entirely preset, and any parameter can be varied "in-process" and with manual override capability for any and all functions. While under control of the programmer, all parameters which are required to be maintained at constant values are held to the following tolerances: (a) time  $\pm 1$  sec, (b) temperature  $\pm 3^\circ\text{C}$ , (c) gas flow rates  $\pm 0.2$  percent control by mass flow controllers. The system is capable of completely automatic "hands-off" operation. Input and output ends of the system shall interface directly, with no additional hardware necessary, into Buffer T or other equivalent wafer transport systems 36 in. in height that have the same geometric configuration. In bringing wafers from the air track to the susceptor at the input and from the susceptor to air track at output, the handling methods do not damage or contaminate the wafers. There is a manual handling option in case of automatic handling malfunction. All necessary sensor outputs are brought out to a common terminal so that these can be used as inputs to a master computer that will assume control of this system and others. Automatic shutdown of the system is provided in case of a malfunction except for those functions which might be necessary to prevent damage.

## B. DISCUSSION

All toxic substances that are emitted from the system are handled in a manner that is not hazardous to personnel and is in accordance with local EPA guidelines and OSHA standards. The system is designed with personnel safety in mind. All materials of construction are durable and, in critical areas, are the highest purity and compatibility available so as not to introduce contaminants into otherwise ultra-pure chemical reactants used. All such materials are specified.

- 1) All plumbing is type 316 stainless steel.
- 2) All tubing has been LOX-cleaned.
- 3) All piping and components meet the requirements of ANSIB 31.1
- 4) Assemblies have been flushed and cleaned in accordance with Class I semiconductor standards.



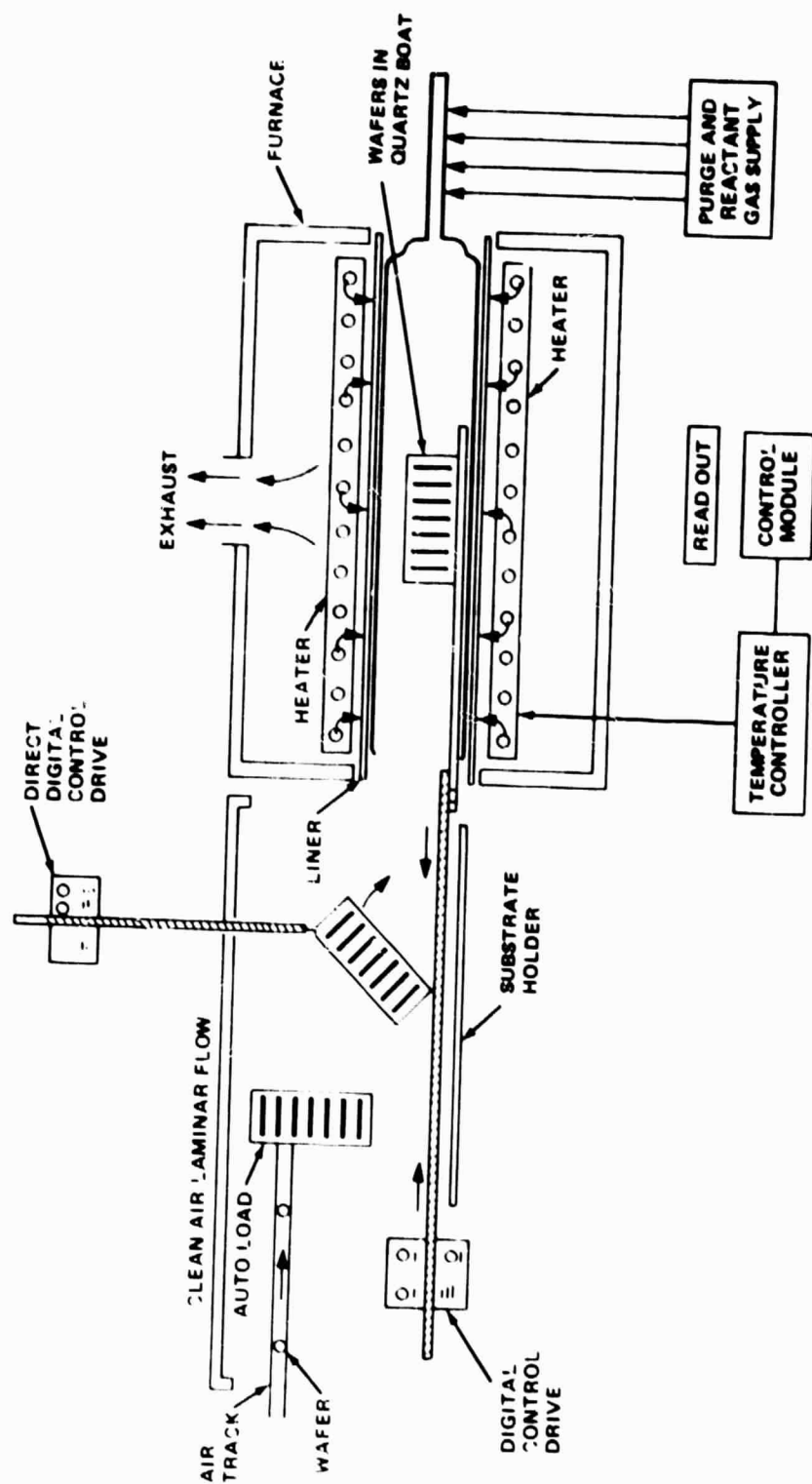


Figure 1. Automatic chemical vapor deposition system.

5) The organization and arrangement of components are solicited for correct gas mixing and ease of maintenance and meet the requirements of ANSI Standard B31.1.

6) All materials in contact with LOX have been certified compatible therewith.

All cabinetry that is subject to the possibility of explosion has been designed to be explosion proof. Maximum dimensions on any one console are 10 ft x 3 1/2 ft x 6 ft 8 in. or 9 ft x 4 ft x 6 ft 8 in. Total The CVD system has been installed and demonstrates that the system will meet the specification. Gas filters and dryers for each source cabinet have been furnished, except where gas is not compatible with the dryer media. They are mounted on the top of the cabinet and plumbed to each panel. The use of this equipment shall appreciably prolong the time between regular maintenance procedures. Silicon dioxide-dopants, polycrystalline silicon, aluminum oxide, and silicon nitride tests have been performed to insure that the chemical vapor deposition system/systems meet the requirements.

1. Hardware Requirements. Hardware requirements are as follows:

(a) Polycrystalline silicon deposition

- |                             |                 |
|-----------------------------|-----------------|
| 1. Thickness range          | 2000 Å - 8000 Å |
| *2. Uniformity              |                 |
| 3. Throughput/hr capability | @ 1000 Å        |

(b) Silicon nitride deposition

- |                             |                |
|-----------------------------|----------------|
| 1. Thickness range          | 500 Å - 1000 Å |
| *2. Uniformity              |                |
| 3. Throughput/hr capability | @ 1000 Å       |

(c) Aluminum oxide deposition

- |                             |                |
|-----------------------------|----------------|
| 1. Thickness range          | 500 Å - 3000 Å |
| *2. Uniformity              |                |
| 3. Throughput/hr capability | @ 1000 Å       |

(d) Silicon dioxide deposition

- |                             |                 |
|-----------------------------|-----------------|
| 1. Thickness range          | 1000 Å - 2000 Å |
| *2. Uniformity              |                 |
| 3. Throughput/hr capability | @ 1000 Å        |

(e) Doped silicon dioxide deposition and undoped

\*Dopant diluent - argon



- |  |   |
|--|---|
| 1. Thickness range                       | 2000 Å - 3000 Å   |
| *2. Uniformity                           |   |
| 3. Dopant concentration range in silicon | $1 \times 10^{16}$ to $1/3 \times 10^{19}$<br>atoms/cm <sup>3</sup> after drive<br>in<br>@ 2000 Å |
| 4. Throughput/hr capability              |   |

\*Uniformity

- Across wafer  $\pm 5\%$
- Within run averages  $\pm 5\%$
- Run to run averages  $\pm 5\%$
- Week to week averages  $\pm 5\%$

2. Reactants and Temperature. A tabulation of reactants and temperature ranges required in chemical vapor deposition system/systems is as follows:

Et	React	Carrier Gas	Reactants	Temperature Range	Dopants*
Si <sub>3</sub> N <sub>4</sub>		H <sub>2</sub>	SiH <sub>4</sub> NH <sub>3</sub> SiH <sub>4</sub>	900°-1100°C	
		N <sub>2</sub>	NH <sub>3</sub>	600°-700°C	
Poly-silicon		H <sub>2</sub>	SiH <sub>4</sub>	850°-1000°C	
		N <sub>2</sub>	SiH <sub>4</sub>	600°-700°C	
SiO <sub>2</sub>		H <sub>2</sub>	SiH <sub>4</sub> CO <sub>2</sub> or N <sub>2</sub> O	600°-900°C	
		N <sub>2</sub>	SiH <sub>4</sub> CO <sub>2</sub> or N <sub>2</sub> O	500°-900°C	
		H	SiCl <sub>4</sub> CO <sub>2</sub> or N <sub>2</sub> O	800°-1000°C	
SiO <sub>2</sub> "N" or "P"		N <sub>2</sub>	SiH <sub>4</sub>	200°-500°C	PH <sub>3</sub> , B <sub>2</sub> , H <sub>6</sub>
Doped or Undoped					

\*Dopant diluent argon

### 3. System Hardware.

a. **Furnaces** - The furnace is a two chamber module with heating chambers, SCR firing circuit, assemblies, excess temperature systems, and wiring. The modules are complete including (1) exhausted enclosure in furnace base for mounting of gas panel, (2) enclosed scavenger at load end with collar for exhausting spent process gases, (3) enclosed scavenger at source end with collar for exhausting tube connection area, (4) heat exchanger for minimizing chamber heat loss to the room, and (5) complete interconnect wiring for computer monitoring and control of furnace temperature. Figure 2 presents the automated CVD system furnaces.

b. **Load Stations** - The load station is designed to mate with the above furnace module. A class 100 clean air provided over the work/load area houses the following: (1) dual overhead florescent lighting with aluminum egg-crate diffuser, (2) perforated stainless steel work deck, (3) two buffer "T" assemblies, (4) boat loaders, etc., and (5) a complete boat transfer mechanism. Figure 3 presents the loading and unloading of CVD furnaces.

c. **Gas Panels** - The gas panels are designed to incorporate the necessary features for process compatibility, control, rangeability, maintainability, and safety.

1) All plumbing is type 316 stainless steel to be compatible with the process gas purity levels necessary and to insure leak-tightness.

2) All panels have been leak-tested to  $10^{-9}$  cc/sec of helium using a mass spectrometer leak detector.

3) Whenever practical, from a servicing point of view, plumbing joints have been welded to eliminate leakage sources and trapped volumes.

4) Plumbing lines are short and direct to minimize purge times.

5) All tubing has been LOX-cleaned, and assemblies have been flushed and cleaned in accordance with Class I semiconductor standards.

6) Wiring has been neatly harnessed and identified for ease of maintenance.

7) Accessory items such as fittings, filters, valves, regulators, gauges, etc., have been chosen for quality and reliability rather than price.

8) The organization and arrangement of components have been selected for correct gas-mixing, safety, process compatibility and ease of maintenance.

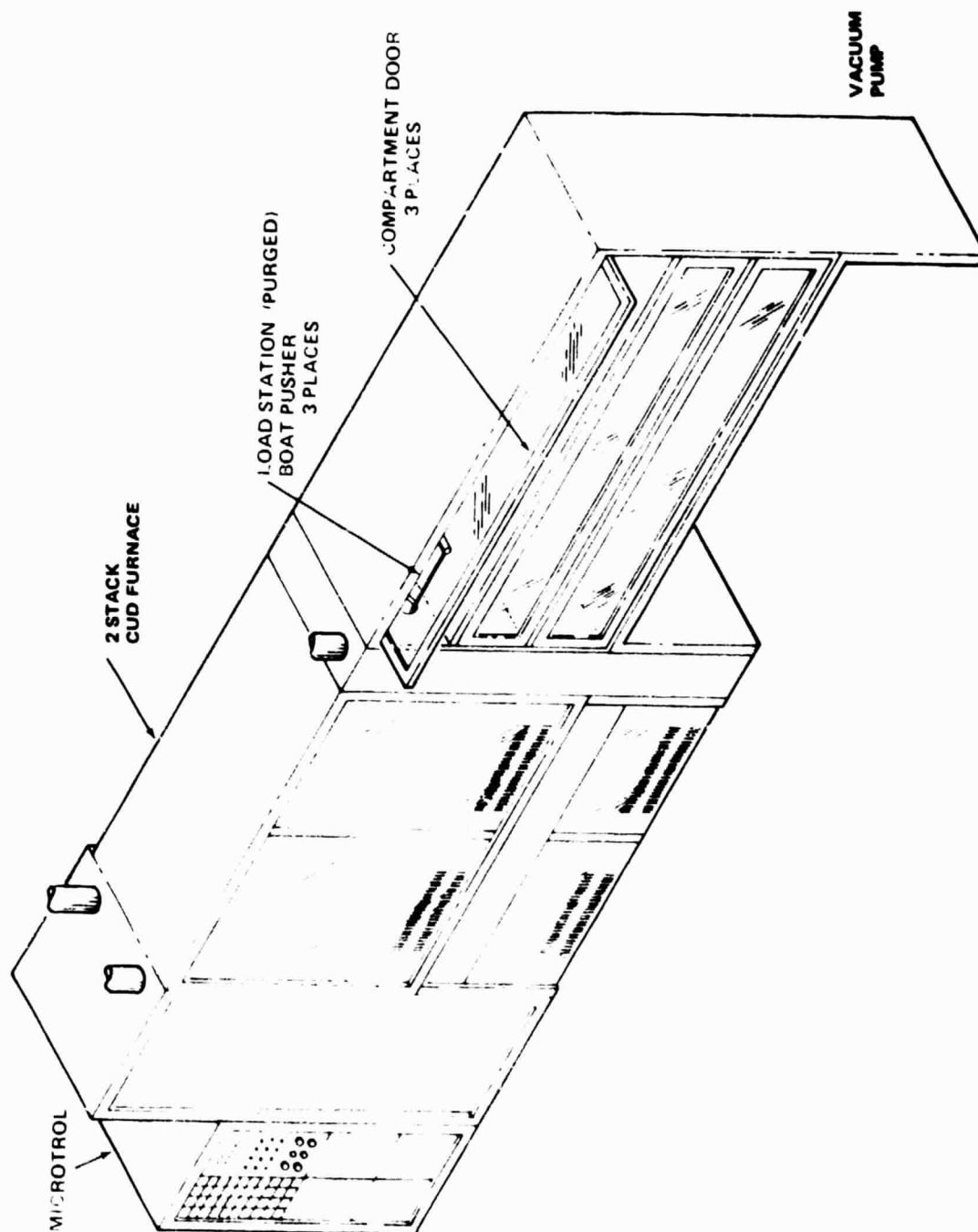


Figure 2. Automated chemical vapor deposition system.

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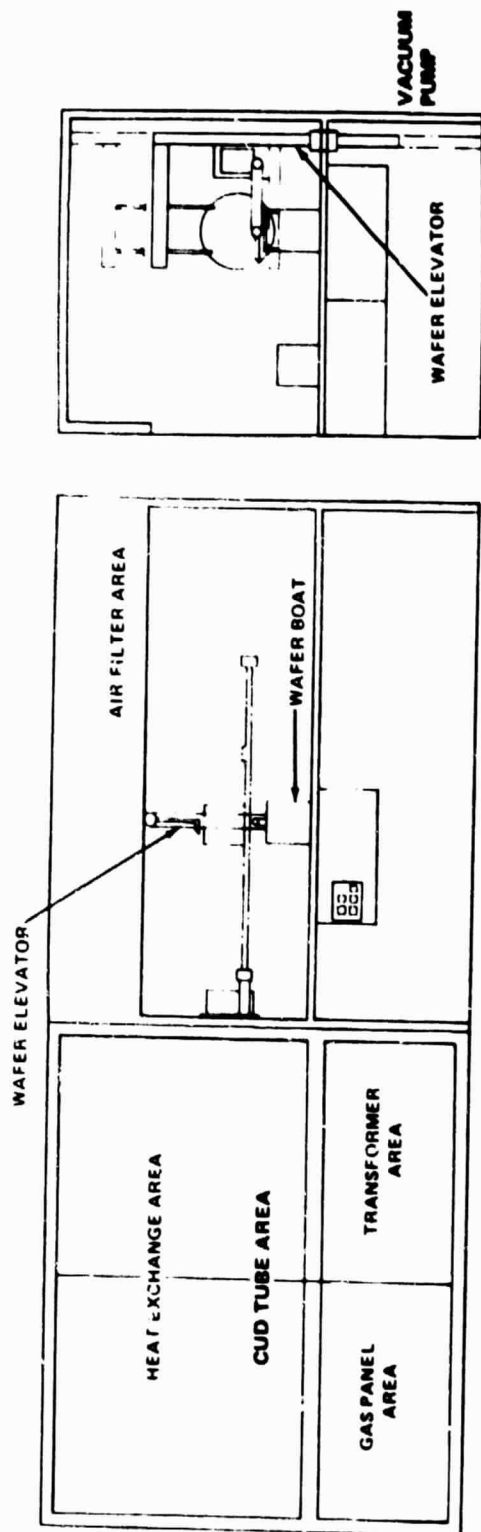
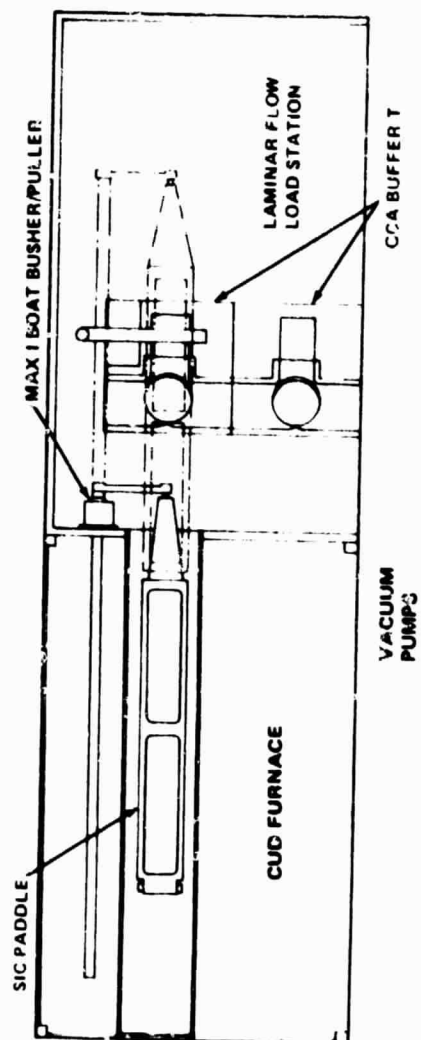


Figure 3. Automated chemical vapor deposition boat pusher/puller system.

9) A manual shutoff valve has been included on each gas circuit to allow system changes or maintenance.

10) The nitrogen flow controller on the panel automatically supplies purge flow for the process tube during power outages.

d. Boat Loader - Wafer boat loader/unloader is used. The boat loader has a stationary support rod mounted in the load station and positioned under the movable rod for greater stability. Speed controls are contained in the controller. Important features are as follows:

1) The drive is a dc motor instead of a stepper or an SCR-controlled ac motor. This has eliminated the problem of vibration which causes crystal dislocation in the silicon wafers at processing temperatures.

2) There is a built-in, adjustable overload clutch for fail safe operation whenever sticking or drag exceeds the preset limit

3) The design is compact and easy to troubleshoot and maintain.

4) Start-stop limit switches are the solid-state, photo-diode type for long life and reliability.

5) The drive mechanism is rack and gear for positive speed control and position.

e. Buffer T - The Buffer T is designed to control the variations of wafer flow in in-line wafer processing systems. The buffer T functions as a three-mode directional air track switch gate and wafer storage medium. These modes are by-pass, load, and unload and are manually selected or remotely commanded from the series control circuits or user supplied logic signals.

f. Transfer Mechanism - Automatic wafer handling includes a transfer mechanism that moves wafers into boats from the Buffer T or air track and inserts them into the furnaces and removes them back to the Buffer T or air track.

g. Direct Digital Controller - A controller with direct digital control is used for control of the two stack furnace. The controller is contained in a single cabinet that is located adjacent to the furnace. Other features are as follows:

1) Communication with a central computer is accomplished through an interface.

2) The system is easily expandable.

3) Parallel manual/automatic control are provided for all functions.

- 4) A highly reliable DEC PDP-8/A computer unit is used.
- 5) The system is completely self-contained and performs all data acquisition and control functions.
- 6) The controller receives instructions from an up-line computer and feeds back information to it. This includes loading of the controller with all the data necessary to carry out a particular process sequence.
- 7) The controller is supplied with a built-in printer to provide hard copy reports on lots run through the furnace. In addition, all faults which occur during a process are printed and simultaneously entered into memory for transmission to the up line computer. Programs and program changes are also printed and transmitted.
- 8) A paper tape reader is built into the operator's panel to enable the rapid input of new programs and to reenter the basic controller executive program in the event it is lost through some catastrophic failure.